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IS 6977 (1973): 1,5-Diaminoanthraquinone, technical [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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SPECIFICATION FOR 1,5-DIAMINOANTHRAQUINONE, TECHNICAL

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110001

Price Rs 5·00

December 1973

AMENDMENT NO. 1 JANUARY 1988

TO

IS:6977-1973 SPECIFICATION FOR 1,5-DIAMINOANTHRAQUINONE, TECHNICAL

[*Page* 4, *Table* 1, *Sl No.* (iv), *col* 3] - Substitute '93.0' *for* '90.0'.

(PCDC 11)

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Indian Standard
SPECIFICATION FOR 1,5-DIAMINOANTHRAQUINONE, TECHNICAL

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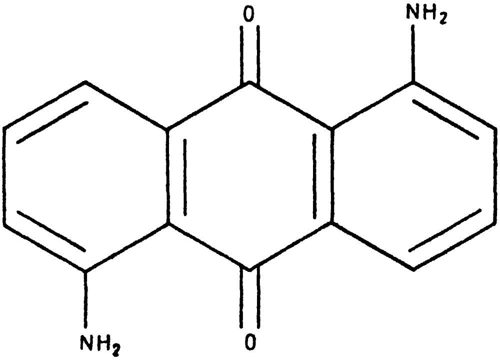
Indian Standard
SPECIFICATION FOR 1,5-DIAMINOANTHRAQUINONE, TECHNICAL

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 12 June 1973, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Chemical Division Council.

0.2 1,5-Diaminoanthraquinone ($C_{14}H_{10}O_2N_2$) is a very important intermediate used in the manufacture of vat and disperse dyes. It has the following structural formula:



1,5-DIAMINOANTHRAQUINONE

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for 1,5-diaminoanthraquinone, technical.

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*Rules for rounding off numerical values (*revised*).

2. REQUIREMENTS

2.1 Description — The material shall be in the form of red-brown powder and free from visible impurities.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR 1,5-DIAMINOANTHRAQUINONE, TECHNICAL

SL No. (1)	CHARACTERISTIC (2)	REQUIREMENT (3)	METHOD OF TEST, REF TO CL No. (4)
i)	Moisture content, percent by mass, *Max*	0·5	A-2
ii)	Sulphated ash, percent by mass, *Max*	4·0	A-3
iii)	Melting point	Shall melt within the range of 3°C including 300°C	A-4
iv)	Assay, percent by mass, *Min*	90·0	A-5

3. PACKING AND MARKING

3.1 The material shall be suitably packed in steel drums lined with polythene or as agreed to between the purchaser and the supplier.

3.2 The containers shall be marked with the following:

a) Name of the material;
b) Tare, net and gross mass of the material;
c) Name of the manufacturer and trade-mark, if any; and
d) Batch number and date of manufacture.

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certfication Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 The material shall be sampled in accordance with **3** of IS : 5299-1969*.

---

* Methods of sampling and tests for dye intermediates.

APPENDIX A

(*Table* 1)

METHODS OF TEST FOR 1,5-DIAMINOANTHRAQUINONE, TECHNICAL

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF MOISTURE CONTENT

A-2.1 Moisture shall be determined by drying in an oven at 100 to 110°C in accordance with the method given under **9.3** of IS : 5299-1969†.

A-3. DETERMINATION OF SULPHATED ASH

A-3.1 Determine the sulphated ash of the sample in accordance with Method B given under **11** of IS : 5299-1969†.

A-4. DETERMINATION OF MELTING POINT

A-4.1 Determine the melting point of the sample in accordance with the method given under **8** of IS : 5299-1969†.

A-5. ASSAY

A-5.0 Outline of the Method — The purity of the sample is checked by employing the chromatographic method. 1,5-diaminoanthraquinone is separated chromatographically and determined quantitatively by spectrophotometer.

A-5.1 Reagents

A-5.1.1 *Alumina* — Aluminium oxide chromatographic grade and of desired activity.

A-5.1.2 *Benzene* — Dried for 24 hours over fused calcium chloride, filtered and then distilled. Portion distilling out between 79°C and 80°C is collected for use.

A-5.1.3 *Acetone* — Dried as above and distilled.

---

*Specification for water, distilled quality (*revised*).

†Methods of sampling and tests for dye intermediates.

A-5.2 Preparation of the Chromatographic Column

A-5.2.1 A glass tube, 40 cm long and having 1·5 cm as its internal diameter, joined with a 50-ml thistle funnel at the upper end and fitted with a stop-cock at the lower end, is set up vertically such that the percolation passing through the column can be collected conveniently in a 250-ml conical flask. Place a cotton-wool plug in the tube and press it to the bottom of the tube by means of a glass rod, flattened latitudinally at the end. Place a disc of filter paper, cut to the approximate internal diameter of the chromatographic tube on top of the cotton-wool.

A-5.2.2 Prepare a slurry of about 15 g of the alumina in benzene, and pour it into the tube. Wash down the sides of the tube and pack the column by lightly tapping the tube. Place first a disc of filter paper and then a cotton-wool plug at the top of the alumina column. Care should be taken to see that the alumina column remains soaked with the solvent, such that at least a 2-cm layer of the excess solvent remains always, on top of the alumina. On no account, shall the alumina be left to dry. In the event of the latter happening, the alumina in the column shall be reslurried and repacked.

A-5.3 Procedure

A-5.3.1 *Preparation of Standard Chromatographically Pure 1,5-Diaminoanthraquinone* — Dissolve 200 mg of a dry and pulverized sample 1,5-diaminoanthraquinone, technical, in 200 ml of benzene. Reflux for half an hour. Cool to room temperature, using a water-bath.

A-5.3.1.1 Transfer the solution prepared in **A-5.2.1** gradually to the chromatographic column prepared with alumina and dry benzene (**A-5.1.2**) as described in **A-5.2**. When all the solution loaded has entered the column, and the cotton at the top of the alumina is just dry, add a small quantity of benzene. Go on adding this benzene in small lots till the cotton gets colourless, and the reddish-orange band of pure 1,5-diaminoanthraquinone just leaves the top of alumina column. Now fill up the column with a mixture of benzene and acetone (95 : 5 respectively). With the slow passage of this mixture through the column the reddish-orange band further subdivides itself into the following bands (from bottom to top) :

Faint red — impurity
Reddish-orange — 1,5-diaminoanthraquinone
Red — 1,8-diaminoanthraquinone
Pink — impurity
Red — at top

A-5.3.1.2 Collect the reddish-orange elute which represents pure 1,5-diaminoanthraquinone. A set of five to six such columns is kept in operation for collecting a sizable quantity of pure material. Filter through filter paper to remove stray alumina particles. Concentrate the total quantity of elute containing 1,5-diaminoanthraquinone by distillation of the eluent until crystallization occurs on cooling to room temperature. Filter out the crystals and dry. The dry and pulverized pure product is further dried under high vacuum at 150 to 160°C.

A-5.3.1.3 In order to check its purity, dissolve about 25 mg (accurately weighed) in 25 ml of dry benzene and pass 10 ml of the cooled solution through a chromatographic column, set up as described in **A-5.2**, eluting with the mixture of benzene and acetone (95 : 5) as necessary. Collect the band carefully (only one band should appear). Make up to 100 ml and determine its optical density at 475 nm. Take 10 ml of the original solution, dilute to 100 ml and determine its optical density in the same way. A difference in the two figures for optical density amounting to more than 0·003, indicates the presence of an impurity. The chromatographic purification shall in such a case, be repeated until this check test is satisfied.

A-5.4 Preparation of the Standard Calibration Graph for the Particular Optical Instrument — Prepare a number of standard solutions of the chromatographically pure 1,5-diaminoanthraquinone (obtained as in **5.2.2**) in dry benzene, varying in concentrations from 0·6 mg/100 ml to 2·0 mg/100 ml, with a difference of 0·2 mg/100 ml between successive concentrations.

Take readings for optical density, or percentage transmittance for the above-mentioned concentrations at a wavelength of 475 nm (*see* Notes) using the specially matched cells specified for the particular spectrophotometer used. The temperature of the solutions immediately before and after the measurements shall be 27 ± 2°C.

Where the instrument is calibrated in percentage transmittance only, the optical density may be read out from a standard table. Plot the calibration curve of concentration (in mg/100 ml) against optical density.

NOTE 1 — Maximum absorption (or minimum transmittance) has been observed at a wavelength of 475 nm by standard instruments. However this may need checking with the particular instrument to be used. Several readings for absorbance using solutions of varying concentrations are taken at different wavelengths and that for maximum absorption determined from the graph.

NOTE 2 — The standard calibration curve and the spectrophotometer shall be checked for accuracy from time to time.

A-5.5 Determination of the Purity of a Sample

A-5.5.1 Weigh accurately about 35·0 mg of a dry and pulverized sample of 1,5-diaminoanthraquinone, technical, in a dry 250-ml quick-fit conical flask. Add about 200 ml of dry and distilled benzene (*see* **A-5.1.2**). Reflux for half an hour, using a water condenser. Cool to room temperature by keeping in water-bath. Transfer the solution of 1,5-diaminoanthraquinone to a dry 250-ml volumetric flask. Rinse the conical flask with small amounts of benzene, two to three times, and transfer the rinsings also to the volumetric flask. Dilute the solution in the volumetric flask up to the mark with dry and distilled benzene, and shake the solution well.

A-5.5.2 Load 10 ml out of the above 250-ml solution of 1,5-diaminoanthraquinone on a solvent-soaked alumina column (dimensions and method of preparation of the column shall be the same as prescribed in **A-5.2**) and chromatograph the material by the method described in **A-5.3.**

A-5.5.3 The reddish-orange elute is collected in a dry 100-ml volumetric flask, and made up to the mark with dry and distilled benzene. The contents are thoroughly shaken. Use this solution *(y)* to take readings for either percentage of transmittance or absorbance.

A-5.5.4 Adjust the wavelength of maximum absorption (λ max) at 475 nm and then adjust the instrument in such a way that the transmittance through the blank is recorded at the 100 percent, reading after inserting the cell with the 'blank' solution (dry and distilled benzene only). Now replace the cell with the solution (*see* **A-5.5.3**) of the sample and read 'percentage transmittance'. Referring to the standard tables for conversion of transmittance to optical density, read out the concentration C against the optical density from the standard calibration graph (**A-5.4**) in terms of mg/100 ml of the final dilute solution.

A-5.5.5 *Calculation*

$$\text{Purity, percent by mass} = \frac{2\,500 \times C}{M}$$

where

C = the quantity of 1,5-diaminoanthraquinone in mg contained in 100 ml of solution *(y)*; and

M = mass in mg of the sample taken.

INDIAN STANDARDS

ON

DYE INTERMEDIATES

IS:	
2630-1973	Nitrobenzene (*first revision*)
2740-1973	Sulphanilic acid, technical (*first revision*)
2741-1973	*β*-Naphthol (*first revision*)
2744-1964	α-Naphthylamine
2833-1973	Aniline, technical (*first revision*)
3229-1973	Naphthionic acid (sodium salt) (*first revision*)
3242-1965	*β*-Oxynaphthoic acid (bon acid)
3562-1965	*n*-Nitrotoluene, technical
4265-1967	4-4′ Diaminostilbene 2-2′ di-sulphonic acid
4334-1967	*o*-Chloroaniline
4335-1967	*m*-Chloroaniline
4336-1967	*p*-Chloroaniline
4425-1967	*p*-Nitrotoluene *o*-sulphonic acid
4523-1968	Acetoacetanilide
4524-1968	Acetoacet *o*-chloroanilide
4525-1968	*n*-Aminoacetanilide
4526-1968	2, 5 Dichloroaniline
4527-1968	2-Nitro-4-chlorotoluene
4528-1968	4-4′-Dinitrostilbene 2-2′ disulphonic acid (disodium salt)
5042-1969	1-Aminoanthraquinone
5043-1969	2-Aminoanthraquinone
5044-1969	Benzanthrone
5045-1969	Metanilic acid, technical
5299-1969	Methods of sampling and tests for dye intermediates
5438-1969	Nitrobenzene-*m*-sulphonic acid, sodium salt
6258-1971	*o*-Nitroanisole
6259-1971	Anthraquinone, technical
6260-1971	*p*-Nitroanisole
6264-1971	J-acid
6265-1971	Quinizarine, technical
6266-1971	1, 4-Diaminoanthraquinone, technical
6961-1973	3-Bromobenzanthrone, technical
6962-1973	3, 9-Dibromobenzenthrone, technical
6977-1973	1, 5-Diaminoanthraquinone, technical

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Printed at Sree Saraswaty Press Ltd, Calcutta-9, India